DEVELOPMENT OF NONMETALLIC FIBROUS MATERIALS FOR PROTECTION OF PERSONNEL FROM

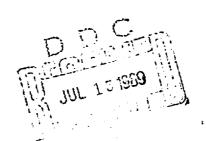
Richard J. Shernit

HIGH INTENSITY THERMAL RADIATION

The B. F. Goodrich Company

November 1968

AIR FORCE MATERIALS LABORATORY
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433



DEVELOPMENT OF NONMETALLIC FIBROUS MATERIALS FOR PROTECTION OF PERSONNEL FROM HIGH INTENSITY THERMAL RADIATION

Richard J. Shernit

FOREWORD

This report was prepared by The B. F. Goodrich Company, Aerospace and Defense Products Division, and covers work performed under Contract AF33(615)-3427. This contract was initiated under Project 5878, "Thermal and Visible Light Protective Screen for Fighter Aircraft". The work was accomplished during the period of 10 January 1966 to 30 September 1968. The contract was monitored by the Air Force Materials Laboratory, with Mr. William Rooney (MAAE) the project engineer.

The B.F. Goodrich project engineer was Mr. Richard J. Shernit of Department 1805, Accessories Engineering. Principal contributors to the technical and production effort were Messrs. K. E. Paige, J. F. Krocheski, and J. W. Warner. The program was under the administrative direction of J. A. Briscoe, Manager, Accessories Engineering.

This technical report has been reviewed and is approved.

√ H. Ross, Chief

Fibrous Materials Branch Nonmetallic Materials Division

Air Force Materials Laboratory

ABSTRACT

This report details the technical development performed to select an inorganic oxide reflective coating which, when combined with a reinforcing fabric and insulative backing, can replace MIL-C-27347 Aluminized Glass for thermal radiation shielding. Magnesium oxide, lanthanum oxide, and titanium dioxide were evaluated as reflectants. Glass and Nomex fibers and fabrics of various weaves were studied. Silicone rubber compounds were developed and modified to yield the proper degree of insulation.

The early stages of the program centered on coating fibers and weaving them into suitably reflective materials. The end product proved unacceptable. Work was then performed to develop a reflective coating which could be applied to a commercially available fabric. After selecting silicone rubber compounds for proper insulation, an acceptable material was used to construct a prototype F-100F shield. Fabrication and cleaning problems showed the end product to be unsatisfactory, although laboratory samples could meet acceptance criteria.

Reflective coatings had thus far been produced by mixing inorganic oxides in a Teflon, potassium silicate, or silane binder. Later work centered on using a silicone rubber binder for the reflective oxides.

A durable end product resulted from these studies. The reflective coating may be sprayed or spread from a RTV (room temperature vulcanizing) compound. The Nomex reinforcing fabric combines flexibility and high temperature resistance. An aluminum filled silicone rubber backing provides a heat sink and the necessary opacity to minimize heat transmitted through the shielding material.

This document is subject to special export controls and each transmittal to foreign government or foreign nationals may be made only with prior approval of the Air Force Materials Laboratory, Wright-Patterson AFB, Ohio 45433.

This page purposely left blank.....

TABLE OF CONTENTS

SECTION		PÂĢÉ
I	INTRODUCTION	1
II	SUMMARY	3.
ĮĨĬ	SIMULATION OF THERMAL RADIATION	5
IV	INORGANIC OXIDES AS REFLECTANTS	8 ;
'V '	PHASE I EVALUATION Introduction Preparation Evaluation of Reflectance and Thermal Radiation Resistance	10 10 11 12
	Discussion of Results - Phase I Evaluation Recommendations of Phase I Evaluation	15 16
VI	PHASE II INITIAL INVESTIGATION Introduction Testing Procedures - Phase II Initial Investigation Results of Phase II Initial Investigation Recommendations of Phase II Initial Investigation	17 17 18 20 25
VII	PHASE II INVESTIGATION Problem Areas Complete Phase II Test Results	27 27 30
VIII	PROTOTYPÉ SHIELD CONŠTRUCTION FROM HS-958	39
IX	SILICONE RUBBER AS A BINDER MEDIUM	40
X	REEVALUATION OF FABRIC AND INSULATIVE SUBSTRATES Nomex Reinforcing Fabric Insulative Backing	S 42 42 43
XI	PROPERTIES OF THE FINAL MATERIAL Introduction Preparation Evaluation	45 45 45
XII	FLAME RESISTANCE OF SHIELD MATERIALS	50
XIII	CONCLUSIONS AND RECOMMENDATIONS	52

This page purposely left blank.....

SECTION I

INTRODUCTION

The explosion of an atomic or hydrogen bomb results in the emission of a shock wave, nuclear particle radiation, and an intense blast of thermal radiation. If the delivery vehicle is manned, these personnel attempt to flee the drop zone so that the aircraft is not damaged by blast shock and the deadly effects of nuclear radiation are minimized.

However, even supersonic aircraft leave these men within the range of high intensity thermal radiation, transmitted as a blinding flash of light and its accompanying searing heat energy.

When the aircraft delivers a nuclear weapon from a low-altitude approach and then escapes at a low altitude, it encounters more severe conditions than those of high-altitude delivery and escape. One of the worst conditions is the fairly common one in which there is total overcast over a snow covered terrain.

The protection of personnel from thermal radiation resulting from a nuclear detonation can be effected only by a shielding material designed to absorb or reflect this energy. Aircrew personnel are presently protected by specially designed hoods. An aluminized glass fabric is generally used as a reflecting barrier.

This material is prone to rapid degradation under flexing, cleaning, and general handling. The reflecting aluminum film rapidly abrades off as a fine powder. This deficiency led to this search for a superior shield material. This material needs to retain its surface integrity after repeated flexing, extended storage, and general maintenance and check-out handling. It must also possess the ultimate property of reflecting the blast energy and preventing a large rise in backside temperature.

Such a material was developed as a result of this contract effort. The reinforcing material is a tightly woven Nomex fabric. On the exterior surface of this shielding material a highly reflective silicone rubber thermal control coating is deposited. The interior surface is a silicone rubber insulation containing aluminum powder to minimize emittance of heat to within the enclosure.

The material is extremely flexible, and only approximately twenty-five thousandths of an inch in thickness. Thus, a shield protecting the entire cockpit of an aircraft can be folded into a small space and then easily extended into position in the event of an emergency.

SECTION II

SUMMARY

It was proposed that composite materials of a high-temperature resistant fabric, coated with a solution containing inorganic oxides, and backed with an insulating layer of silicone rubber would provide the necessary protection and yet remain flexible and durable after repeated usage and handling. The inorganic oxides possess excellent reflectance properties, and a sufficient coating applied to the base fabric material will reflect a high percentage of the blast energy. The base fabric, itself, should be resistant to high temperatures. Nomex and glass fabrics were suited for investigation. The silicone rubber backing combines insulative properties with flexibility and durability at high and low temperature extremes.

Potassium silicate and Teflon were proposed as binders for adhesion of the reflective oxide to the base fibers.

Many candidate materials underwent the examination of pertinent physical characteristics performed during Phase I of the contract. Ten sample types were then subjected to an initial investigation during Phase II. Four superior samples were then selected. Adhesion problems prompted the development of a new coating system. A commercially available, silane-type, primer/adhesive was substituted as the binder. This system was used to equal the performance of aluminized glass.

The total thickness of the composite material, however, was .035" vs. .018" for aluminized glass. Since it can be shown (Reference 1) that 2.0 calories/cm² is a tolerable limit for total heat transmittance, the requirement was changed from 1.5 cal./cm² to 2.0 cal./cm². A .025" thick composite was produced which met acceptance criteria.

A prototype F-100-F thermal shield was then constructed from this material, designated HS-958. Deficiencies of the material became apparent during fabrication and subsequent handling. Some flaking of the reflectant surface was experienced during fabrication, and the surface was nearly impossible to clean when soiled, especially with oils and greases.

Attention was then turned to use of silicone rubber as the binder for the reflectant exide. A high temperature resistant, continuous coating was thus provided. The surface is highly resistant to abrasive degradation and easy to clean with soap and water or a mild solvent. Combined with a Nomex reinforcing fabric and insulation layer of silicone rubber, the final material meets or exceeds MIL-C-27347.

The recent availability of Nomex fabrics woven from 100 denier fiber also made it possible to reduce the thickness of the composite material. Since the smaller diameters of the 100 denier fibers make a tighter weave possible, the Nomex reinforcing layer acts as an improved barrier to the transmission of the thermal energy. Coupled with the silicone rubber reflectant coating, a material thickness of .025" limited the total heat transmitted through the material during an exposure to less than 1.5 cal./cm².

However, military demands for Nomex materials limited commercial availability of 100 denier materials during the contract period. It was thus possible to fabricate only a portion of the ten F-100F shields required by contract from 100 denier fabric. A standard 200 denier fabric was substituted after the small supply of improved fabric was depleted.

The 200 denier material was also qualified to meet or exceed MIL-C-27347 with the exception of thickness. A .028" thickness of the alternate material is required to limit heat transmittance to 1.5 cal./cm².

SECTION III

SIMULATION OF THERMAL RADIATION

The quartz lamp bank utilized in materials testing duplicates a specified thermal flux which is equivalent to that of a thermonuclear detonation under certain conditions. The actual number of calories per square centimeter which is being delivered to a sample is determined by calorimetric means, and can be varied by changing the lamp-to-sample distance.

The literature discloses that the maximum energy density of a fusion detonation lies in the near ultraviolet (3000 to 4000 Å) during the initial energy pulse, then shifts to a maximum in the visible range during the secondary pulse. Actual incident radiation spectrum depends upon the nature of the subject to fireball air path. Computation of the qualitative aspects can be performed if atmospheric characteristics such as pressure, humidity, and path length are given for a specific yield weapon.

When considering the photon energy of incident radiation, we note that energies in the visible and near infrared regions of the spectrum cause only thermal agitation when striking a material. The relationship $E = h \times f$, where E is the photon energy, h is Planck's Constant, and f is frequency, shows the higher energies occur at higher frequencies. The higher frequencies of ultraviolet light, X-rays, and gamma rays cause higher photon energies capable of causing molecular interactions, ionization, etc.

For a nuclear detonation occurring in the atmosphere, ultraviolet energy is appreciably absorbed by the atmosphere, and only visible and near infrared affects are considered in the scope of this work on thermal radiation shields. It is not as important to consider a specific frequency of peak energy as it is to consider that the applied energy distribution occurs in the visible and near infrared since the lower frequencies of these waves cause only the generation of heat in a material.

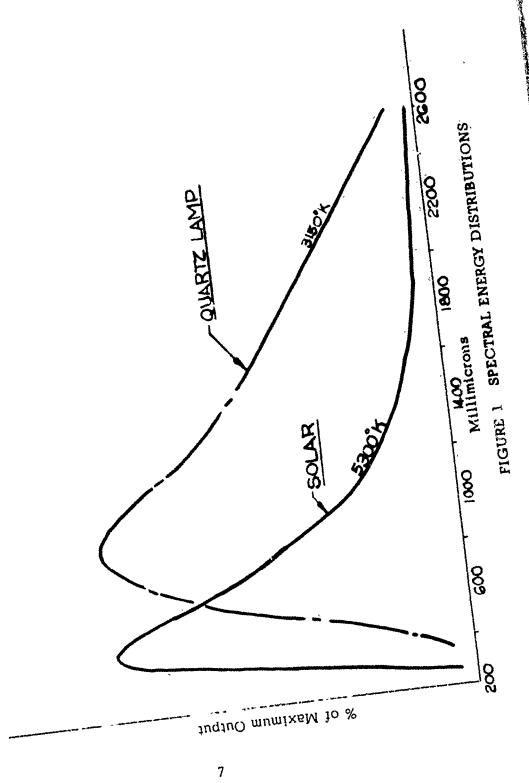
In further discussions, it will be assumed that a medium yield nuclear detonation is well approximated by the solar spectrum occurring outside of the earth's atmosphere. Due to atmospheric absorption of ultraviolet energies, only the visible and near-infrared regions are considered.

The graph of spectral energy distribution (See Figure 1) shows the relative energies versus wavelengths for solar and quartz lamp radiation. The solar spectrum possesses a color temperature of 5800°K, peaking at 500 millimicrons.

The quartz lamp bank peaks at approximately 910 millimicrons, with a color temperature of 3150°K. General Electric 1000T3 quartz lamps were used in an overloaded condition. Overloading the lamps at 440 volts results in a shift of the peak output from its normal value of 1160 millimicrons. This results in a closer approximation of the solar spectrum.

The main difference between the solar and quartz lamp spectrums is the position of the peak energy output. Forty percent of the solar energy falls in the visible and forty-six percent in the near infrared. Fifteen percent of the quartz lamp energy falls in the visible and seventy percent in the near-infrared. Both outputs act only to generate heat in the material they strike.

Since the reflectance of an inorganic oxide loaded material is essentially constant (at approximately 80%) in the visible and near infrared regions, the qualitative simulation of the blast spectrum is not important. Thus, the quartz lamp bank provides adequate simulation of the thermal energy delivered to the aircraft.



SECTION IV

INORGANIC OXIDES AS REFLECTANTS

The most direct approach in minimizing the heat build-up in a shielding material is to maintain the lowest possible temperature at the front side, reflecting surface.

The temperature gradient between the front and backside surfaces is then held to a minimum during an exposure to thermal radiation. Therefore, heat transmitted through the material by conduction is also minimized.

Radiant energy falling upon an opaque surface will be absorbed () or reflected (). The law of conservation of energy requires that the sum of these fractions be equal to unity or:

$$\propto + \rho = 1$$

To determine the temperature of this system, it is necessary to also know the emissivity of the material (e). This property relates the emitting characteristics of real bodies to an ideal body. A portion of the energy absorbed is emitted, the unemitted fraction then raising the kinetic energy level of the substance and hence providing a "temperature". Using the Stefan-Boltzman law:

$Q_B = 6 AT^4$	Q_B = Radiation from a block body
and	A = Surface Area of body
anu	T = Temperature
$Q_G = e \delta A T^4$	Qg = Radiation from actual body
$e = \frac{Q_B}{Q_G}$	6 = Stefan-Boltzman Constant

¹Total emissivity of a surface is the ratio of the rate of heat loss by radiation alone from that surface compared to that from a perfect radiator at the same temperature.

The surface temperature (T) of a body due to incident radiation is given essentially as:

$$T^4 \sim K \frac{\approx}{e}$$

where K = f(I, 6) and I is the incident flux.

Thus, the temperature of the reflecting surface is directly proportional to the ratio of absorption to emittance. An aluminized surface, such as found on MIL-C-27347 aluminized glass, exhibits an emittance of 0.06 (Ref. 2). An inorganic oxide coating was similarly shown to exhibit an emittance value of 0.80.

Inorganic oxides such as magnesium oxide, lanthanum oxide, and titanium dioxide (and also an aluminized surface) reflect 80 to 90% of radiation in the visible and near infrared ranges. Thus, taking the absorbence value of both inorganic oxide and aluminized surfaces as 0.15, the following ratios result:

Inorganic Oxide Surface: $\frac{4}{e} = .15/.80 = 0.19$

Aluminized Surface: $\alpha/e = .15/.06 = 2.50$

The lower \(^{\omega}/e\) ratio of inorganic oxides, and therefore their ability to minimize the surface temperature, led to their investigation under this contract.

SECTION V

PHASE I EVALUATION

INTRODUCTION

Nomex and glass fibers and fabrics were chosen for the base materials due to their inherent resistance to high-temperature degradation. These materials were coated with a silane primer so that adequate adhesion of the reflectant solution could be obtained.

The reflectant solution was then applied to the primed base material. Magnesium oxide and lanthanum oxide were investigated as reflectants. These inorganic, nonmetallic materials exhibit high reflectivity and emissivity values. Their low absorbtivity-to-emissivity ratios yield lower temperature increases during exposure to radiation.

Potassium silicate or a Teflon/water dispersion was used as the binder. A small amount of molybdic acid (H2MoO₄) was also added to the reflectant solution. The use of a complexing agent such as molybdic acid for the suppression of absorption peaks was found to increase reflectance by approximately 5% in the range of 1500 to 2000 millimicrons. Since the end product must maintain flexibility when using a water-based binder, complete dehydration must not occur. The residual water will then absorb radiation. But the use of a small amount of molybdic acid was found to suppress this absorption. A small increase in reflectivity in this range becomes important when it is considered that a relatively high percentage of radiant energy occurs in this band. It was also noted that the slightly acidic solution which results gave improved physical properties to the solution so that more uniform coatings were obtained.

Unimats of the coated fibers were made by winding three layers on small, 1/8" thick, hardboard or aluminum squares. Mats of each sample were then submitted for reflectance measurements and testing for resistance to thermal radiation.

 $6" \times 6"$ samples of coated fabrics were made for evaluation. Additional pieces were made so that effects of laundering on reflectivity and thermal exposure resistance could be studied.

PREPARATION

Beta-diameter glass fibers, 200 denier Nomex, and 200 denier rayon were coated with a slurry of inorganic oxide and Teflon or potassium silicate binder in a water base. The fibers were dipped in the slurry, passed through a 9 foot heated tunnel at 375°F to dry, and wound onto spools at rates of up to 120 feet per minute. The slurry was made by mixing the given oxide, binder, and solvent in a Waring blender.

Early samples were not primed before passing through the slurry.

After varying proportions of the ingredients, the following formulation was found to exhibit the best physical properties after application to the fiber:

30 grams binder
70 grams reflectant oxide
600 grams water
0.5 grams molybdic acid (for absorption suppression)

Relatively poor adhesion of the reflectant coating indicated that priming of the fiber was necessary. A commercially available, silane type, primer/adhesive (Chemlok 607, supplied by Hughson Chemical Company, Erie, Pa.) was used.

The fiber was primed and dried during an initial pass through the coating equipment. The primer was an equal mixture of Chemlok 607 and anhydrous alcohol. Three passes through the reflectant solution and drying tunnel were then necessary to build up a continuous layer of reflectant on the fiber. The results of reflection and thermal radiation testing for the various combinations of fiber, reflectant, and binder are given in Table I.

Glass, Nomex, and rayon fabrics were brush coated with a slurry of reflectant oxide, binder, and solvent prepared as above. The same formulation of slurry was found to be optimum for coating the fabrics.

The fabrics were first primed by dipping in an equal mixture of Chemlok 607 and anhydrous alcohol. After drying, three brush coats of the reflectant solution were necessary to build up an adequate coating. The solution was allowed to dry between coats. The results of reflectance and thermal radiation testing for the various combinations of fabric, reflectant, and binder are given in Table II.

EVALUATION OF REFLECTANCE AND THERMAL RADIATION RESISTANCE

Reflectivity Measurements

A Beckman DS-2 spectrophotometer with a MgO integrating sphere was used to determine reflectance over the range of 260 to 2200 millimicrons. It was assumed the detonation spectrum is closely approximated by the solar spectrum occurring outside the atmosphere. The reflectance curves were then integrated with known data of the energy distribution of solar irradiance. Thus, final reflectance calculations yielded results relative to an incident spectrum resulting from a nuclear detonation.

The total spectrum was divided into three ranges:

Ultraviolet	260-400 millimicrons	8.82% Total Energy
Visible	400-700 millimicrons	40.14% Total Energy
Near Infrared	700-2200 millimicrons	46.40% Total Energy

The ultraviolet energy is small and appreciably absorbed by the atmosphere. Only results of visible and near infrared reflectance were tabulated for these samples.

The results given for percentage of reflectance are averages of calculated reflectances in the visible and near infrared ranges. The minimum required percentage of reflectance was 80% for any material selected for further evaluation.

Thermal Radiation Resistance

A simple lamp bank was used to expose the samples to a flux of radiant energy. Sixteen 1,000 watt quartz infrared lamps (GE 1000T3) were staggered in a double tier arrangement.

The bank was operated at 277 volts. A color temperature of 2500°K results from these conditions. A ferrotype-tin curved reflector was used to reflect the back emission of the lamps. The intensity of the radiant flux vs. lamp-to-sample distance was roughly determined by calorimetric means.

A simple calorimeter was constructed by depositing carbon soot from an acetylene torch on one end of a 1" diameter copper rod. Two small holes were drilled 1" apart along the length of the rod. Thermocouples were inserted into these holes so that the temperature gradient could be determined. A stream of water was passed over the back surface of the rod to provide a constant temperature heat sink. The blackened end of the rod absorbed essentially 100% of the incoming heat energy, and knowledge of the value of thermal conductivity of copper permitted calculation of the incoming thermal flux after temperature equilibrium was reached.

The samples were then tested at 6 calories/cm² sec. for periods of 10 seconds. Unimats of coated fibers were clamped to a back-up plate of high temperature resistant transite board, and coated fabrics were taped with silicone-glass tape to the back-up plate.

Coated Fibers

The following combinations of materials were designated for investigation.

Reflectant	<u>Binder</u>	Base Fiber
Magnesium Oxide	Teflon	Nomex
Magnesium Oxide	Potassium	Nomex
	Silicate*	
	w/Molybdic Acid	
Lanthanum Oxide	Potassium Silicate	Nomex
Lanthanum Oxide	Potassium Silicate w/Molybdic Acid	Nomex
Magnesium or	Teflon or	Glass (Betà
Lanthanum Oxide	Potassium Silicate w/or without	diameter)
	Molybdic Açid	

^{*}For this combination molybdic acid was required to obtain good adhesion.

In addition to the required materials, nearly all possible combinations were fabricated and investigated. This work was done to insure a thorough study of the constituent materials. Rayon fiber was also coated with two of the reflectant solutions.

The following nomenclature will be used throughout to designate the type of coated fiber or fabric:

<u>Fiber or Fabric</u>	Binder	Reflectant
N Nomex	T Teflon	M MgO
G Glass	K Potassium Silicate	L La ₂ O ₃
R Rayon		

Thus, a NTM type signifies a Nomex fiber or fabric, Teflon binder, and magnesium Oxide reflectant.

TABLE I

Туре	Vis. and N.I.R. % Reflectance	Resistance to Thermal Exposure
RTM	84	Poor
RKM	82 ⁻	Poor
NTM	86	Excellent
NTL	(Poor adhesion of	coating to fiber)
NKM	85	Excellent
NKL	86	Good
GKM	84	Excellent
GKL	81	Good
GTM	90	Cond
GTL	88	Gros

Ceated Fabrics

All combinations of base fabric, binder, and reflectant who prepares are slowed. RTM type tabric had been made during a previous development centrally from their rayon fiber, woven into fabric. This fabric was available and we such their meanther results of woven fiber and coated fabric techniques. (Screen solutions that taining both magnesium and lanthanum oxides were also prepared and study A. The glass, rayon, and Nomex fabrics possessed the following physical characteristics:

	Glass	Payon	Nonch
Thread Count (Warp)	57	80	101
Thread Count (Fill)	54	68	93
Torsile Strength (Warp)	300	120	260
Tensile Strength (Fill)	230	120	247
Thickness (Inches)	.013	.009	.010
Weight (oz./yd. ²)	8.9	4.5	o. ₫

All coated fabric samples underwent identical testing as performed on fiber samples. In addition, the fabrics were laundered in a mild detergent solution. All physical characteristics were then compared.

TABLE II

TADBE II				
Summary of Test Results - Coated Fabrics				
Vis. and N.I.R. % Reflectance Resistance Type % Reflectance After Laundering Thermal Ex				
RTM	82	80	Poor	
NTM	82	82	Excellent	
NTL	ITL (Poor adhesion of coating to fabric)			
NTL/M	(Poor adhesion of coating to fabric)			
NKM	82	82	Excellent	
NKL	82	82	Good	
NKL/M	86	82	Excellent	
ĢTM	86	86	Poor	
GTL	GTL (Poor adhesion of coating to fabric)			
GKM	86	-86	Good`	
GKL	84.	84	Pŏor	
			,	

DISCUSSION OF RESULTS - PHASE I EVALUATION

Nomex fiber or fabric was found to be the superior base material. While reflectance values of coated Nomex were slightly lower than those of coated glass, Nomex withstood exposure to thermal radiation with no evidence of scorching.

Coated glass was frequently scorched to a small extent after only one exposure to the lamp bank. In addition, glass materials inherent brittleness indicated that the desired flexibility could not be attained. Coated rayon materials were always badly scorched or burned during exposure.

Reflectance values of coated fibers are, on the whole, higher than those of coated fabrics. But, the testing on the RTM materials showed that normal weaving processes tend to loosen and brush off small amounts of the reflective coating. Hence, comparable reflectance values would be expected from coated fabrics and coated fiber, woven into material.

Both Teflon and potassium silicate showed promise as binders. Magnesium oxide appeared to be the better choice of reflectant due to superior resistance to thermal exposure and laundering, and in providing more workable solutions for application to the base material. Use of a combination of reflectants showed that slightly higher reflectance may be attainable by adding a small amount of lanthanum oxide to a MgO solution.

RECOMMENDATIONS OF PHASE I EVALUATION

Evaluation showed that NKM and NTM type fiber or fabric were the superior materials. Consideration of reflectivity and resistance to thermal exposure, laundering, abrasion, and flexing indicated that fiber coating or fabric coating techniques would yield a comparable final product.

SECTION VI

PHASE II INITIAL INVESTIGATION

INTRODUCTION

It was required that two systems of reflectant coating be applied to two weaves of Nomex material and backed with silicone rubber (for a tôtal of four sample types). This number of samples were then to be subjected to the complete Phase II evaluation.

Four types of woven fiber samples and six types of coated fabric samples had been selected as superior. This total of ten sample types was necessary for a complete investigation of the coating and fabrication techniques which were under study.

Conducting the required series of tests on each of these ten sample types would have increased the duration of the Phase II evaluation greatly. It was decided that an initial investigation of each of the ten sample types would be conducted. The most critical parameters were studied, and the superior sample types (four were selected from this study) were then subjected to the complete Phase II evaluation.

The Phase II Initial Investigation was conducted on the samples listed below. Three already-woven fabrics and two weaves of coated fibers were studied. Two types of reflectant solution were applied. One solution used potassium silicate as the binder, and the other used a Teflon water dispersion. The samples were backed with .005" of a titanium dioxide pigmented silicone rubber. The samples were vacuum cured at 300°F for 30 minutes and post cured for four hours at 400°F. (Coated fabric samples were coated after the initial cure.)

TABLE III

List of Samples - Phase II Initial Investigation

Sample No.	Designation	Type	Weave
1	NTM-P	Woven Fiber	Plain
2	NKM-P	Woven Fiber	Plain
3	NTM-T	Woven Fiber	Twill
4	NKM-T	Woven Fiber	Twill
5	NKM-1	Coated Fabric	Plain
6	NTM-1	Coated Fabric	Plain
7	NKM-45	Coated Fabric	Satin
8	NTM-45	Coated Fabric	Satin
9	NKM-47	Coated Fabric	Twill
10	NTM-47	Coated Fabric	Twill

The dash numbers (-1, -45, and -47) refer to the three candidate Nomex fabrics. The fabrics were HT-1, HT-45, and HT-47 (Stern and Stern Textiles Code Numbers).

TESTING PROCEDURES - PHASE II INITIAL INVESTIGATION

Reflectance

A Beckman DK-2 spectrophotometer with a MgO integrating sphere was used to determine the % reflectance exhibited by the samples. Minimum reflectance in the visible and near infrared of 85% was a target goal.

Abrasion

Abrasion testing was performed using the apparatus diagrammed below. Two fabric surfaces were abraded together. The abrasion tester was run at 90 cycles per minute, and the specimens were under a 2 pound load. The materials were to be superior to the Aluminized Glass Control Standard and show less than 2% loss in reflectance after testing.

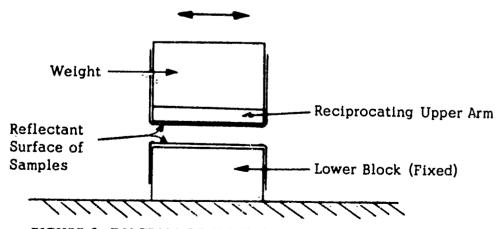


FIGURE 2 DIAGRAM OF ABRASION TEST APPARATUS

Thermal Radiation Resistance

Materials were to withstand an exposure to radiant energy of 40 cal./cm² @ 15 cal./cm²/sec. with less than 2% loss in reflectance after testing. The 16 lamp quartz lamp bank was used as the source of thermal radiation.

Heat Transmittance

Materials were exposed to the conditions listed below as produced and after abrasion testing. Insulative properties were to be superior to those of the control standard. The 16 lamp quartz lamp bank was again used as the source of thermal radiation.

Three exposures at each condition listed below were run:

60 cal./cm 2 @ 6 cal./cm 2 /sec. 40 cal./cm 2 @ 15 cal./cm 2 /sec. 36 cal./cm 2 @ 20 cal./cm 2 /sec.

During and after all exposures, the material was not to give off fumes which were toxic or smoke which would irritate the eyes or restrict visibility. Also, no greater than a total of 1.5 cal./cm² was to be emitted from the backside surface or the material during any exposure.

A calorimeter was not yet available to accurately determine heat energy emitted from the backside surface of the sample. It was first assumed that MIL-C-27347 meets the heat transmittance requirement (Ref. 3). A simple calorimeter was then constructed to determine the relative heat output of samples to the aluminized glass control standard. A 1" diameter copper disk (.125" thick) was mounted 1/4" behind the samples. One side of the disk was blackened with carbon soot from an acetylene torch to provide an absorbing surface for the transmitted heat. A thermocouple was silver soldered to the center of the opposite side. The voltage output of the thermocouple was monitored with a Honeywell M100-120 Electro-Magnetic Damped Subminiature Galvanometer and recorded on a Honeywell Model 906A-1 Visicorder (recording oscillograph).

Abrasion/Flex

This test was designed to simulate the abrasion, flexing, and creasing of the shield fabric under actual operating conditions. It has been noted that MIL-C-27347 fabrics begin to lose the all minum coating after 25 cycles of opening and refolding a canopy shield into its stowed position. Sharp creasing of these fabrics always disturbs the aluminum surface coating.

The abrasion tester was modified for this test. The sample was folded around a small diameter rod as shown in the figure below.

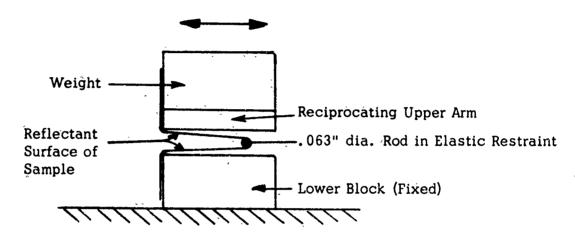


FIGURE 3 DIAGRAM OF ABRASION/FLEX TEST APPARATUS

The reciprocating motion of the upper arm rolls the sample back upon itself over the rod. The resultant abrasion, flexing, creasing, and rolling of the specimen subjects it to conditions similar to those encountered by a shield in actual usage. The moving arm was weighted with two pounds for these tests.

RESULTS OF PHASE II INITIAL INVESTIGATION

Reflectance

Preliminary data showed the % Reflectance of NTM-1, NKM-1, NTM-45, NTM-47, and NKM-47 to be in the range of 80-83%. More data was needed to determine an average value of reflectance for each material. The absolute maximum reflectance would be near 85% due to the roughness of the fabric surfaces. The control standard, Aluminized Glass Fabric, exhibited reflectance near 90%. This correlated test procedures well with published data on this material.

The woven fiber samples (NTM-P, NKM-P, NTM-T, and NKM-T) had been processed by Prodesco, Inc., Perkasie, Pa. Fifty thousand feet of NTM type fiber and fifty thousand feet of NKM type fiber (all 200 denier Nomex) had been coated. One pass through the primer and three passes through the reflectant slurry were again necessary for an adequate coating. A 24" x 24" sample of the four types of woven fibers listed below were produced.

TABLE IV

Woven Fiber Physical Properties			
Designation	Weave	Width	End Count
NTM-P	Plain	24"	72 x 72
NKM-P	Plain	24"	72×72
NTM-T	2/1 Twill	24"	72×72
NKM-Ť	2/1 Twill	24"	72×72

Much of the reflectant coating was lost during the weaving operation. The materials exhibited approximately 75% reflectance. The materials utilizing Teflon as a binder (NTM-P and NTM-T) withstood the weaving operation with less loss of reflectant coating.

The results of this testing showed the first six sample types mentioned to be the most promising.

Abrasion

All tests were run for 2,500 cycles, at which point damage to all materials was visibly evident. The specimens were then compared with regard to the extent of damage. Abraded samples were also subjected to Heat Transmittance Tests.

The visual examination of the abraded samples is summarized in the following chart.

Abrasion Test Results

Phase II Initial Investigation

Sample Type		Description of Damage
Aluminized Glass	(Control Standard)	Aluminum surface abraded off the high surface of the weave.
NTM-1 .	(Coated Fabric)	Partial loss of MgO coating to bare fabric.
NKM-1	(Coated Fabric)	MgO surface abraded off the high surfaces of the weave.
NTM-45	(Coated Fabric)	Partial loss of MgO coating to bare fabric.
NKM-45	(Coated Fabric)	Considerable loss of MgO coating.
NTM-47	(Coated Fabric)	Partial loss of MgO coating.
ŃKM-47	(Çoated: Fabric)	MgO surface abraded off the high surfaces of the weave.
NTM-P and NKM-P	(Woven Fiber)	Slight fraying of fibers and loosening of weave.*
NTM-T and NKM-T	(Woven Fiber)	Slight fraying of fibers and loosening of weave.*

^{*}These materials are coated Nomex fibers, woven into fabric. All exhibited increased stiffness over commercially available bare fabrics. No loss of MgO coating was noted after abrasion, but much of this coating had been lost during the weaving operations.

As a result of this testing, it was concluded that the coated fabric samples should be investigated further. Materials utilizing the Teflon binder lost MgO by rolling off small particles of this coating. These particles increased in size as they rolled over more coating and, in most cases, small areas of bare fabric were exposed. Materials using the potassium silicate binder lost MgO by physically shearing off layers of this coating.

Thermal Radiation Resistance

The BFG Quartz Lamp Bank facility was used to subject all materials to 45 cal./cm² @ 15 cal./cm²/sec. No visible damage was noted for any of the ten sample types or the Aluminized Glass Control Standard. No smoke or fumes were noted during these exposures.

Heat Transmittance

All materials were exposed to the following conditions:

60 cal./cm
2
 @ 6 cal./cm 2 /sec. 45 cal./cm 2 @ 15 cal./cm 2 /sec.

The 20 cal./cm²/sec. exposure rate was not obtained for these tests.

Test results were similar for all NTM and NKM materials. The designation NXM is used on the graphs to show typical results for any material. (The total thickness of all materials tested was .015", .010" Nomex fabric plus .005" white silicone rubber insulation).

Figure 4 shows the results of these tests. Materials were tested "as produced" and after abrasion testing. No single material proved superior, and all materials showed excessive heat transmittance when compared to SRGA fabric.

Abrasion/Flex Tests

The condition of the samples after a specified number of cycles is listed in Table VI.

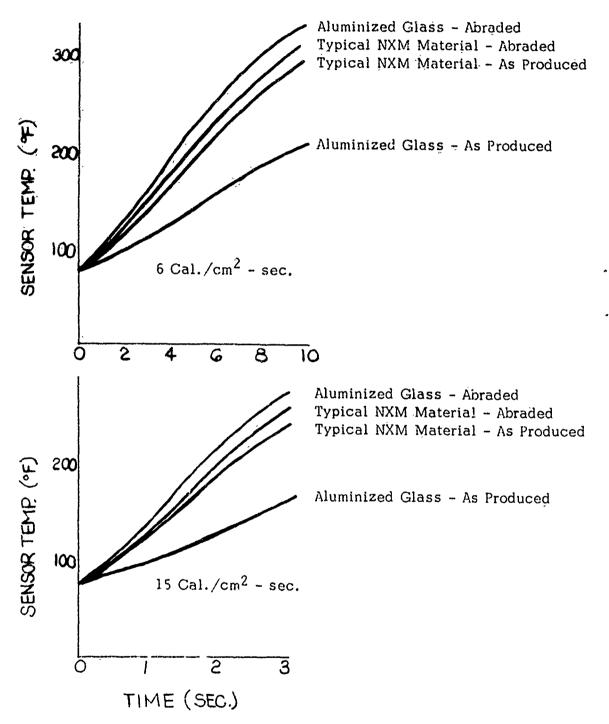


FIGURE 4 HEAT TRANSMITTANCE DATA PHASE II INITIAL INVESTIGATION

TABLE VI

Abrasion/Flex Test Results Phase II Initial Investigation Sample Type No. of Cycles Description of Damage Aluminized Glass 10 Cracking and loss of aluminum coating: Aluminized Glass 60 Destruction of sample. Complete separation of glass fabric and silicone rubber backing. NKM-1 600 Moderate loss of MgO coating. NKM-45 600 Moderate loss of MgO coating. NKM-47 600 Slight loss of MgO coating.

Slight fraying of fibers.

Fabric frayed and weave loosened.

The woven fiber samples (NKM-P and NKM-T) showed less durability under test. Results are not given for the sample types using a Teflon binder. Preliminary Abrasion/Flex tests on these materials showed the same type of microscopic rolling-off of the reflectant coating that was noted after linear abrasion. This type of material was eliminated due to extensive loss of reflectant.

600

600

RECOMMENDATIONS OF PHASE II INITIAL INVESTIGATION

NKM-P

NKM-T

This evaluation showed the woven fiber samples to be stiffer, less durable, and to possess a lower percentage of reflectance. The HT-45 samples, when processed, were similar to the HT-1 materials. The greater availability of HT-1 fabric was the deciding factor in this case. The four materials selected, then, were:

NKM-1

NTM-1

NKM-47

NTM-47

Problems noted with the NTM and NKM materials after abrasion gave rise to further experimentation with the systems use for application of the MgO coating. Two new sample types were developed and were included in the complete Phase II evaluation in addition to the four noted above. Excessive heat transmittance was noted, and the necessary development work continued on this aspect of material performance.

SECTION VII

PHASE II INVESTIGATION

PROBLEM AREAS

The five tests conducted during the Initial Investigation pointed out problem areas which had not been anticipated earlier in the program. Excessive heat transfer was noted during Heat Transmittance testing, and flaking off of the MgO coating after abrasion and creasing was evident.

Loss of MgO Reflectant Coating

NTM and NKM type coatings had been considered superior during the Initial Investigation. However, these coatings were found to flake off somewhat after sharp creasing and, therefore, were not considered satisfactory. Flaking was not extensive enough to lower the percentage of reflectance measurably.

The Teflon and potassium silicate binders had been mixed with MgO in a water solution. Adhesion of these coatings was found to be dependent on the humidity during preparation and application.

A new coating system was then developed. The dispersion medium was changed to alcohol and the binder used was Chemlok 607, an adhesive supplied by Hughson Chemical Company. This system yielded coatings with exceptionally small particle sizes.

The optimum formulation of the new reflectant coating system was found to be the following:

1000 g. Anhydrous Alcohol

20 g. MgO

0.2 g. Molybdic Acid

2.0 g. Chemlok 607

Smooth, continuous coatings resulted from the use of the new system (designated NM). The percentage of reflectance was not changed in the "as produced" condition. Reflectance after abrasion and flexing was improved.

The NM coating required a curing period, but this was done during the normal post cure required for the silicone rubber backing.

Initially, the Nomex fabric was primed by dipping in a 3 to 1 solution of anhydrous alcohol to Chemlok 607. The fabric was then backed with the silicone rubber insulation and vacuum cured at $300^{\circ}F$ for 30 minutes. Three brush coats of the reflectant solution were then applied. The composite material was then post cured at $325^{\circ}F$ for 8 hours. Higher post cure temperatures resulted in a "yellowing" of the reflectant surface. This was traced to a change in color of the Chemlok 607 primer.

Test results of NM materials are included with those of NTM and NKM materials.

Excessive Heat Transmittance

Inorganic oxide coated materials had been backed with .005" white silicone rubber (total material thickness .015"). The energy transmitted was much in excess of the specification limit. It had been thought that this .015" material would yield adequate results. Basically, two reasons were found for its shortcomings.

- 1. The white silicone rubber was optically transparent to some percentage of heat energy in the visible range.
- 2. Although approximately 80% reflectance was obtained, the fabric transmitted perhaps 15% through openings in the weave. Thus, the silicone received a large amount of thermal energy.

Because the aluminum coated reflective surface of MIL-C-27347 aluminized glass is continuous, there is no direct transmission to the insulating layer. The Nomex fabric, woven from 200 denier fiber, was the least porous available. Thus, development of a silicone rubber compound to absorb the heat was started.

First, the thickness of white silicone was increased until the total material thickness was .035". Heat transfer was still excessive when compared to aluminized glass.

Then, a black, low-conductivity, high optical density silicone rubber underwent modification. Compounds were loaded with iron oxide to lower the transmission in the optical range and with glass frit to scatter and absorb heat energy. When this type of compound was used, a total material thickness of .035" provided results comparable to aluminized glass fabric.

Continuing attempts were made to reduce the thickness of the material. Reducing the material thickness to .025" gave heat transmittance approximately 50% above specification limits.

(A thin, .005", layer of the white silicone had been included between the Nomex fabric and the black silicone insulation. This was done so that the fabric surface would not be darkened by any strike-through of the black compound).

The extended investigation pointed out the following properties which are desirable in a thermal shield material.

- 1. The reflecting layer should provide a high percentage of reflectance and high coefficient of emittance to yield a low equilibrium temperature at the surface.
- 2. The reflecting layer or the fabric substrate should be continuous so that direct transmission of energy to the insulating layer is eliminated.
- 3. The insulating layer should be opaque to essentially all wavelengths of incident thermal radiation.
- 4. The backside surface of the insulating layer should have a low coefficient of emittance so that the minimum amount of heat is re-radiated.

The two properties above which contributed the greatest problem areas were (2) and (4). The front-side surface cannot be made continuous using a semi-porous fabric substrate. Thus, the silicone rubber insulation had to be loaded and thickened to absorb both energy absorbed by and transmitted through the reflecting layer.

Resolution of Heat Transmittance Requirements

A meeting was held at W/P AFB on 8/17/66 to discuss the progress which had been made in the Phase II evaluation. It was then decided that a thinner, more flexible shield material would be desirable. It was agreed that a total heat transmittance of 2.0 cal./cm² might be tolerated if the material thickness could be reduced to .025" and the weight could be reduced to 25 oz./yd.².

Since no immediate improvement could be made to the reflectant coating or Nomex fabric substrate, the insulative silicone rubber backing was changed to three, .00% plies to improve its opacity and to lower the emittance (£) of the backside surface. Apply of silic me rubber was loaded with iron oxide (black) for opacity and glass for the scattering and absorption of any transmitted radiation. Changing the lackside surface from a black material to a ply of titanium drovide leaded silicone rubber (white) lowered the value of £ for this surface. (The literatus, discloses the value of £ for a black silicone rubber to be approximately 0.9 while £ for a white silicone rubber is approximately 0.6). The composite termal shielding material was given the code number HS-958 and was constructed as follows:

The MgC coated Demon from whe backer with .00% white silicone to prevent strike-through of the black comments. The middle layer of .005" black silicone was loaded with glass trit and the first to provide scattering of radiation and to provide an optically dense layer. The backside layer of .005" white silicone provided good insulative audits a and a low coefficient of crittance.

COMPLETE PHASE II TEST RES TAR

The following test results to so denote to tellow the process of elimination which led to the optimum material. Testing included the four materials which were selected from the Phiso II initial investigation. As testing proceeded, new materials had to be developed to a content deficiencies which were discovered or discussed above. Inferior materials were eliminated as testing proceeded. The purely quantitative data; weight, thickness, etc., is given only for the materials which were still under consideration acar the end of the testing period.

Abrasion Resistance

The following materials were examined for aprasion resistance:

Muminize, Glass NKM-1 NTM01 NKM-47 NTM-47 NM-1 (.035" thickness) NM-47 (.035" thickness) HS-958 The specification required that the materials be supérior to the control standard and exhibit less than a 2% loss in reflectance after test.

A discussion of the resistance to abrasion of aluminized glass, NKM-1, NTM-1, NKM-47, and NTM-47 is given in the section on the Phase II Initial Investigation. The loss of reflectant coating of the NKM and NTM materials led to the development of the NM coating system. This coating was applied to HT-1 and HT-47 Nomex. Although NM-1 and NM-47 showed no improvement in reflectance before abrasion, it was visually noted that fine particles of reflectant coating were not being lost as before.

Figure 5 shows an increase in reflectance during the early stages of the abrasion test (especially in the case of aluminized glass). This was due to a polishing effect (the surface was smoothed out and surface coatings of oil or dirt were abraded away). Later, the reflectant coating was abraded off and the reflectance fell rapidly.

All of the MgO coated fabrics showed superior resistance to abrasion than did aluminized glass. NM-47 was selected as the optimum material on the basis of the minimum amount of MgO coating which was lost.

Reflectance (Materials as produced)

The percentages of reflectance given in the following table are averages of the visible and near infrared reflectance. Values were calculated by integrating total reflectance curves with the solar energy distribution in this range of the spectrum. These values are averages of many samples of each type.

TABLE VII

Reflectance of Materials - Phase II Investigation		
Material	% Reflectance	
Aluminized Glass	86	
NTM-1	82	
NKM-1	82	
NTM-47	82	
NKM-47	81	
NM-1	80	
NM-47	81	
Instrument Error = ±2%		

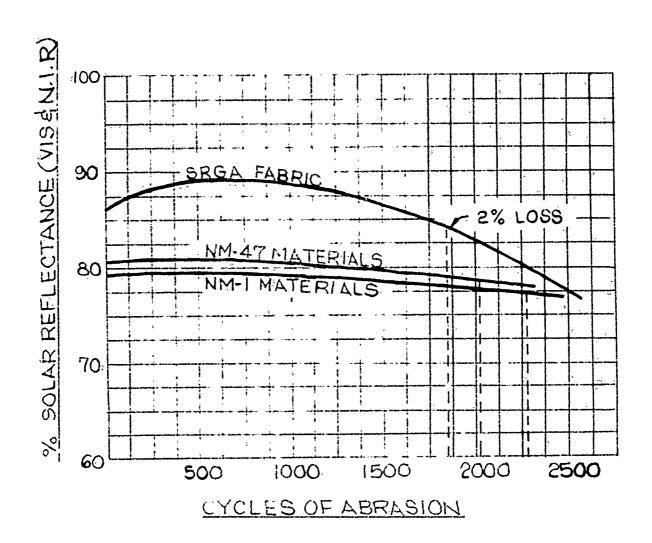


FIGURE 5 ABRASION TEST RESULTS PHASE II EVALUATION

None of the materials showed a marked superiority to the others. The target goal of 85% reflectance was not met, because the roughness of the fabric surfaces presents a limiting factor in this application.

Abrasion/Flex Test

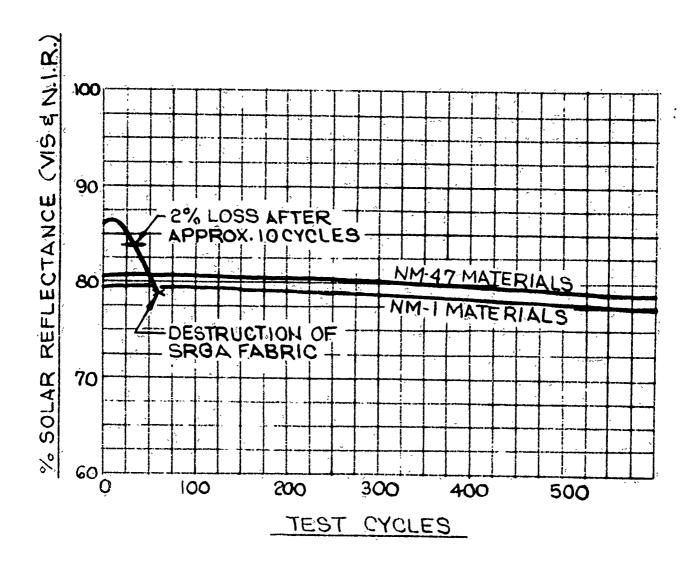
The test apparatus used is described in Section VI. The arm was weighted with a 2 pound load, and the apparatus was operated at 90 cycles per minute.

Aluminized glass fabric showed a 2% loss in reflectance after approximately 10 test cycles and was destroyed after 60 cycles. The new materials showed little damage, even after as many as 1,000 test cycles. (See Figure 6). The lower curve is the average response of NM-1. The upper curve is the average response of NM-47 and HS-958. A 2% loss in reflectance was not observed until after a minimum of 600 test cycles.

Thermal Radiation Resistance

A new source of thermal energy was available for this testing. A Model 5208 High Density Radiant Heater, manufactured by Research Incorporated, Minneapolis, Minnesota, was used. The unit employs six tungsten-filament tubular quartz lamps. A polished metal reflector provides a uniform rate of energy over a 4" by 10" area. General Electric 1000T3 quartz lamps (1,000 watts) were used to provide 6 cal./cm²/sec. General Electric 2000T3 quartz lamps (2,000 watts) were used to provide 15 and 20 cal./cm²/sec. The lamps were operated at 440 volts to provide a closer approximation of the solar spectrum.

The thermal energy was monitored by calorimeters manufactured by Hy-Cal Engineering, Santa Fe Springs, California. The electrical response of the calorimeters is in the order of milliseconds. A Model C-1300-A-15-072 unit was mounted 1/4" behind a sample to determine the rate of transmitted heat energy. A Model C-1300-A-120-072 unit was mounted to the side of the sample at the same level as the reflecting surface to determine the thermal output of the radiant heater. The millivolt output was monitored by a Honeywell M100-120 Galvanometer and recorded on a Honeywell Model 906A-1 Visicorder (recording oscillograph). Calibration charts provided with the calorimeters enabled energy rates to be determined by comparison with their voltage output.



C

Samples $(4-1/2" \times 4-1/2")$ were positioned below the radiant heater (see Figure 7). The samples were held in position by steel needle-points to minimize heat transfer to the surrounding apparatus. The edges of the samples were held down by a 1/4" wide frame of transite hardboard. This prevented curling of the sample and exposure of edges or backside material.

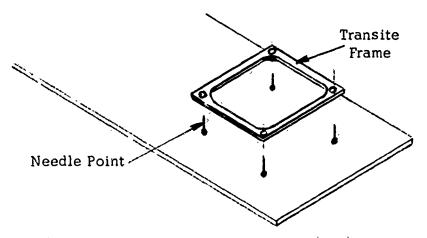


FIGURE 7 SAMPLE POSITIONING FOR THERMAL TESTING

The center areas of 4-1/2" x 4-1/2" samples were abraded. These areas were then directly above the 1" diameter calorimeter used to measure transmitted heat.

Early tests showed that fumes were being given off by the silicone rubber insulation. The amounts of curing agents were changed, and the length of the post curing period was increased. These modifications eliminated the fuming.

All the materials under consideration withstood thermal exposures of 40 cal./ cm^2 @ 15 cal./cm²/sec. No visible damage or measurable loss in reflectance was noted. None of the materials displayed a marked superiority to the others.

Heat Transmittance Test Results

The radiant heater was used to obtain the following exposures. The prescribed duration of exposure at each condition is also given.

 Incident Energy Rate	Duration
6 cal./cm 2 /sec. 15 cal./cm 2 /sec.	10 sec.
15 cal./cm ² /sec.	2.7 sec.
20 cal./cm ² /sec.	1.8 sec.

Aluminized glass fabric, NM-47 (.035"), and HS-958 were investigated. Aluminized glass and NM-47 (.035") limited heat transmittance to less than 1.5 cal./cm 2 . HS-958 limited total heat transmittance to 2.0 cal./cm 2 .

The three materials were subjected to the above exposures after undergoing the Abrasion Test and Abrasion/Flex Test.

After Abrasion, aluminized glass fabric was found to transmit approximately 20% heat energy in excess of the specification limit. NM-47 (.035") and HS-958 showed a negligible increase in heat transmittance. All materials were tested after 2,500 cycles of linear abrasion.

After only 25 cycles of the Abrasion/Flex Test, aluminized glass fabric showed an increase of approximately 50% in heat transmittance. After 600 test cycles, NM-47 (.035") and HS-958 showed only a negligible increase.

This testing showed NM-47 (.035") and HS-958 to meet specification requirements. These materials exhibited far greater durability than did aluminized glass.

Although the stiffness measurements of NM-47 and HS-958 were greater than MIL-C-27347, these materials were sufficiently flexible for the F-100-F radiation shield design.

Thickness

<u>Material</u>	Thickness	
Aluminized Glass	.015 inch	
NM-47	.035 inch	
HS-958	.025 inch	

Weight

Material	Weight	
Aluminized Glass NM-47 (.035")	14 ounces/yd ² 31 ounces/yd ²	
HS-958	23 ounces/yd ²	

Stiffness

Material	Stiffness'		
	Warp	Fill	
Aluminized Glass NM-47 (.035")	.0130 in-lbs	.0130 in-lbs	
Fabric to Fabric	.0240_in-lbs	.0260 in-lbs	
Silicone to Silicone HS-958	.0216 in-lbs	.0247 in-lbs	
Fabric to Fabric Silicone to Silicone	.0122 in-lbs	.0166 in-lbs	

Breaking Strength

Material	Breaking Strength	
	Warp	Fill
Aluminized Glass	150 lbs/in	150 lbs/in
NM-47 (.035")	410 lbs/in	365 lbs/in
HS-958	400 lbs/in	350 lbs/in

Tearing Strength

Material	Tearing Strength	
	Warp	Fill
Aluminized Glass	3 lbs	3 lbs
NM-47 (.035")	32 lbs	24 lbs
HS-958	30 lbs	20 lbs

Cold Crack

This testing was performed in accordance with paragraph 4.6.1.1 of MIL-C-27347 (USAF). No evidence of cracking, stiffening, flaking, or separation of coating was noted.

Materials were tested for % reflectance and heat transmittance after undergoing the Cold Crack test. No changes in the properties of the materials were observed.

Blocking

This testing was performed in accordance with Method 5872 of CCC-T-191b. Eight inch square specimens of aluminized glass, NM-47 (.035"), and HS-958 were examined.

No blocking was observed. The cloth surfaces were free in all cases, and no separation of coatings was in evidence.

Light Transmittance

Aluminized glass fabric, NM-47 (.035"), and HS-958 were examined. No visible light was transmitted during Heat Transmittance exposures.

SECTION VIII

PROTOTYPE SHIELD CONSTRUCTION FROM HS-958

Sufficient HS-958 was processed for the construction of the prototype F-100-F radiation shield to fit a pilot's protective hood package, Part No. 243-53090-31 FSN1680-620-5477.

Individual segments were butted together and joined both inside and outside with 1" tapes. The inside tapes were 1" bias cut HT-47 Nomex, and the outside tapes were 1" bias cut HT-47 backed with uncured .005" white silicone. One inch HT-47 fabric attachment tabs were positioned on the inside surface of the shield.

This assembly was vulcanized on a fiberglass reinforced building form. The MgO reflectant solution was then applied, and the shield assembly was then post cured. Snap fasteners were then located at the proper positions and the edges of the shield material were trimmed to size.

The problem areas are detailed below:

- 1. All edges of the fabric showed extensive ravelling. This included body fabric, attachment tabs, and wire guide loops.
- 2. Several areas showed poor coating of the MgO reflectant. This problem was attributed to excessive handling and residual oils which cannot be totally removed.
- 3. Some small areas showed poor adhesion of the silicone rubber to the Nomex fabric. This problem was again attributed to contamination of the fabric surface.
- 4. Techniques of cleaning the shield fabric proved unsatisfactory. Oil and grease were difficult to remove. Solvents dissolved the grease, but before it could be wiped away the grease penetrated into the pores of the fabric. Thus, discolorations remained after this process is used. Dirt particles could be washed away with a soap solution, but an all-purpose cleaning method was not determined.

SECTION IX

SILICONE RUBBER AS A BINDER MEDIUM

Since only laboratory samples of previous materials were qualifiable, and since the fabrication of a shield from such materials was not practical, attention was turned to the composition of the reflecting surface. As noted previously, the properties desired in this surface are:

- 1. Continuity
- 2. High Reflectance
- 3. High Emittance

Using Teflon, potassium silicate, or Chemlok 607 binders, it was not possible to build up a continuous reflecting layer without experiencing flaking of the surface. Room temperature vulcanizing silicone rubbers were then spread or sprayed on HT-47 Nomex so that the character of such a surface could be investigated.

It was determined that smooth coatings of silicone rubber could be built up on a fabric surface with thicknesses from .001" upward. Such a coating was easy to clean with soap and water or any mild solvent. Loading clear RTV's (room temperature vulcanizing silicone rubbers) with inorganic oxide pigments for white coloration is standard practice, and magnesium oxide, lanthanum oxide, and titanium dioxide exhibit the desired properties of reflectivity and emittance. After attempts were made to pigment clear RTV's with the above inorganic oxides, it was determined that only titanium dioxide pigmentation would produce a highly reflective, flexible surface. Magnesium and lanthanum oxide loaded compounds were brittle after their reflectivity came into the 80-85% range desired.

The superior compound, a white, titanium dioxide loaded, one part RTV silicone rubber exhibited the following properties:

TABLE VIII

	PROPERTIES OF REFLECTIVE RTV COMPOUND		
	Reflectance	83%	
	Emittance	. 87	
	Specific Gravity	1.54	
	Solvent Base	Heptane	,
	Viscosity, CPS	650	
	Loading	Titanium Dioxide	
ų			

The reflective RTV compound was applied to samples by using conventional paint spraying equipment. One and one-half mils of coating was built up in each pass. The coating needed 1-1/2 hours of drying time between passes to eliminate "orange-peeling" of the surface. This time was needed to expel the volatile contents at normal room temperatures.

After a .005" thickness was built up, the surface was highly opaque, and the surface texture of the underlying fabric substrate was not visible. The reflective coating of samples was contaminated with dirt, oils, and greases. In all cases a mild solvent, e.g. alcohol, or simply soap and water cleaned the surface to its original state.

The coating was applied to a metal surface in which a thermocouple had been embedded so that its maximum temperature limitations could be determined. The sample was then placed under the radiant heater. A temperature of 575°-600°F was reached before deterioration of the reflectant surface.

SECTION X

REEVALUATION OF FABRIC AND INSULATIVE SUBSTRATES

NOMEX REINFORCING FABRIC

The commercial availability of Nomex fabrics woven from 100 denier fiber prompted a comparison study of these fabrics vs. the standard 200 denier fabrics. A tightly woven 100 denier fabric, HT-190 (Stern and Stern Textiles Code) was compared to the standard HT-47.

TABLE IX

,		
PROPERTIES OF 100 A	ND 200 DENIER NOMEX FA	ABRICS
5	100 Denier	200 Denier
Designation	HT-190	HT-47
Thread Count (Warp)	118	103
Thread Count (Fill)	78-1/2	75
Tensile Strength (Warp)	133.3 lbs./in.	257.0 lbs./in.
Tensile Strength (Fill)	99.0 lbs./in.	182.5 lbs./in.
Tear Strength (Warp)	10.0 lbs.	27.3 lbs.
: Tear Strength (Fill)	7.0 lbs.	18.5 lbs.
Thickness	.0055 in.	.0101 in.
Weight	2.60 oz./yd. 2	4.94 oz./yd. 2

Samples of these fabrics were backed with .015" of the 3 ply silicone rubber insulation. A .005" coating of the reflective RTV silicone was sprayed on the front surface of the fabrics. The heat transmittance properties of the two composites were then compared during exposures to 6 cal./cm 2 -sec. under the radiant heater.

The transmittance of heat energy through the composite materials was determined to be essentially equal. This testing showed that the reinforcing fabric does little to present a barrier to thermal energy. Direct transmission of heat through the interstices of the weave minimizes the insulative capabilities of the reinforcing fabrics.

Use of a 100 denier fabric resulted in a direct reduction of material thickness of .003"-.005". The weight was also reduced by approximately 1.5 ounces/yd.2.

INSULATIVE BACKING

Using the white, reflective RTV coating with 100 or 200 denier fabrics and the 3-ply insulative coating resulted in heat transmittance levels 15-20% in excess of the aluminized glass control standard. For 100 and 200 denier fabric, the total thicknesses of the shield material composites were .025" and .030", respectively. Such materials met the requirement of limiting heat transmittance to 2.0 cal./cm² during an exposure. However, calendering the 3-ply insulative backing presented processing difficulties. Working with three, .005" layers of uncured silicone gum made the cost of manufacture prohibitive.

From the experience thus far gained, it was decided the desirable properties of an insulative backing were:

- 1. Resistance to high temperatures
- 2. High specific heat
- 3. Low emissivity
- 4. Ability to spread heat through the material to prevent "hot spots" near front surface (relatively high thermal conductivity).

Use of aluminum powder as a filler for the silicone rubber resulted in a relatively high thermal conductivity. The emissivity of aluminum powder is less than 0.1, and when combined with silicone rubber a lower emissivity was expected than that obtained with the previous inorganic oxide loaded compound.

After backing samples with the heat-curable aluminum-loaded silicone rubber, an immediate improvement was noted. This type of insulation made it possible to equal the heat transmittance of aluminized glass with total thicknesses of .025" for 100 denier fabrics and .028" for 200 denier fabrics. The preparation and physical properties of these superior materials are delineated in the following section.

SECTION XI

PROPERTIES OF THE FINAL MATERIAL

INTRODUCTION

Two final materials were prepared and evaluated. The basic material was given the code number HS-966. HS-966 Type I refers to a shield material using a 100 denier base fabric, and HS-966 Type II refers to a material based on a 200 denier fabric.

PREPARATION

To gain adequate adhesion of the silicone rubber compounds to the base fabric, the fabrics were first dipped in a solution of Dow Corning 2260 primer in naptha. After drying for a minimum of one-half hour at room temperature, a .005" layer of titanium dioxide pigmented, heat-curable silicone rubber was calendered on one side of the fabric.

A.015" layer of aluminum-filled silicone rubber insulation was then calendered over the white layer of silicone. (The white silicone was needed only to gain adequate adhesion of the insulative layer to the reinforcing fabric.) This composite was calendered in widths from 18 to 20 inches.

This semi-complete material was then vacuum cured for 30 minutes at 300°F. After cooling, a .005" layer of the reflective RTV silicone coating was sprayed onto the fabric surface. This composite material was then post cured in a circulating air oven for 24 hours at $400^{\circ}F$ for complete removal of volatiles.

EVALUATION

Abrasion Resistance

HS-966, Types I and II, were abraded for 2,000 cycles at 90 cycles per minute under a 2 lb. load. At this point, aluminized glass showed a 2% loss in reflectance. Both types of HS-966 showed no loss of reflectant coating after this testing.

The gloss of the spray finish had been lost, but no appreciable loss in % reflectance could be determined.

Reflectance

The percentages of reflectance for the visible and near infrared ranges are compared below:

Aluminized Glass	86%
HS-966, Type Í	83%
HS-966, Type II	83%

Abrasion/Flex Resistance

Aluminized glass showed a 2% loss in reflectance after 10 test cycles and was destroyed after 60 cycles. HS-966 showed no damage after 60 cycles, and no loss of reflectance could be determined.

Thermal Radiation Resistance

Materials were exposed to 45 cal./cm² at 15 cal./cm² sec. under the radiant heater. HS-966 samples and aluminized glass withstood the exposure with no damage or measurable loss in reflectance.

Heat Transmittance

The radiant heater was used to obtain the following exposures.

Incident Energy Rate	Duration
$6 \text{ cal./cm}^2 \text{ sec.}$	10.0 sec.
15 cal./cm ² sec.	2.7 sec.
20 cal./ cm^2 sec.	1.8 sec.

HS-966 samples and aluminized glass were given three exposures to each of the above conditions. No degradation of the materials was noted during this testing.

The rate of transmitted heat energy vs. time is shown in Figure 8. Only one curve is shown since the performance of HS-966 Type II, HS-966 Type II, and aluminized glass was comparable.

Samples which had undergone 2,000 cycles of linear abrasion also were tested as above. Aluminized glass showed an increase in transmitted heat energy after abrasion. Transmission was approximately 50% in excess of that shown in Figure 8. No change in the performance of HS-966 samples was noted.

Thickness

Material	Thickness
Aluminized Glass	.018"
HS-966 Type I	. 025"
HS-966 Type II	. 028"
HS-966 Type I	. 025"

Weight

Weight
14 ounces/yd ²
23 ounces/yd ²
26 ounces/yd ²

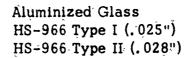
Stiffness.

<u>Material</u>	Warp Fill		
Aluminized Glass HS-966 Type I	.013 inlbs.	.013 inlbs.	
HS-966 Type II	.020 inlbs.	.024 inlbs.	

Stiffnace

Breaking Strength

<u>Material</u>	Warp	<u>Fill</u>
Aluminized Glass	150 lbs./in.	150 lbs./in.
HS-966 Type I	200 lbs./in.	175 lbs./in.
HS-966 Type II	400 lbs./in.	350 lbs./in.



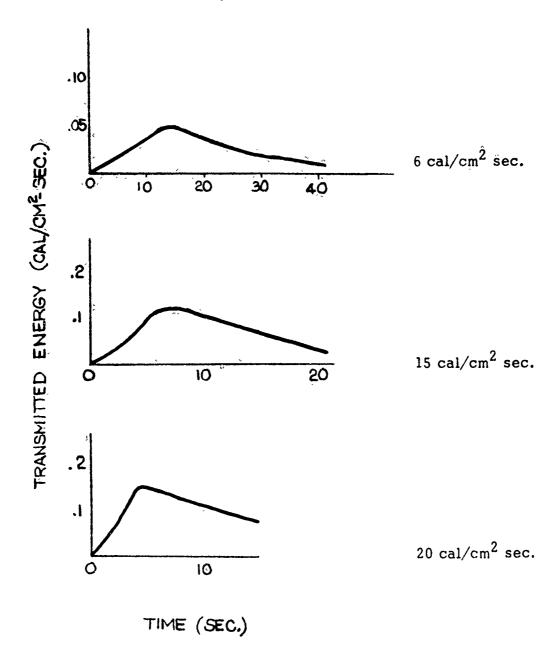


FIGURE 8 HEAT TRANSMITTANCE RESULTS

Tearing Strength.

Material	Warp	Fill
Aluminized Glass	3 lbs.	3 lbs.
HS-966 Type I	12 lbs.	10 lbs.
HS-966 Type II	30 lbs.	20 lbs.

Cold Crack

After testing in accordance with paragraph 4.6.1.1 of MIL-C-27347 (USAF), no evidence of cracking, stiffening, flaking, or separation of coating was noted.

Materials (HS-966 Type I, HS-966 Type II, and aluminized glass) showed no change in reflectance or heat transmittance properties after undergoing the Cold Crack Test.

Blocking

HS-966 Type I, HS-966 Type II, and aluminized glass were tested in accordance with Method 5872 of CCC-T-191b. No blocking was observed. The reflective surfaces were free in all cases, and no separation of coatings was in evidence.

Light Transmittance

No visible light was transmitted during heat transmittance exposures. The optical density of all materials was found to be greater than 4.0.

SECTION XII

FLAME RESISTANCE OF SHIELD MATERIALS

The flame resistance of MIL-C-27347 Aluminized Glass and the newly developed HS-966 were evaluated by following the procedures of Federal Specification CCC-T-191b. Material samples were supported by a three-sided frame with inside dimensions of 2" x 13". One 2" side of the frame was open, and it was at this area that the flame was applied. A gas flame was adjusted to a height of 1-1/2" for the testing.

Samples were first subjected to a horizontal flame test per Method 5906 of CCC-T-191b. The samples were lowered 3/4" into the flame. The duration of the exposures was 15 seconds. The samples were then removed from the flame, and flame time, glow time, and char length were measured.

Other samples were subjected to a vertical flame test per Method 5902 of CCC-T-191b. Samples were lowered 3/4" into the flame. The duration of the exposures was 12 seconds. The samples were then removed from the flame, and flame time, glow time, and char length were measured. This data is presented in Table X.

TABLE X

Material	laterial Horizontal Flame Test			Observations
F	Flame Time (seconds)		Char Length (inches)	
Aluminized Glass (Front Side)	11	0	0. 9	No blistering. Only silicone rubber coating damaged. White ash residue.
Aluminized Glass (Back Side)	8	0	0.8	Same as above.
HS-966 (Front Side)	20	2	0.8	Very little propogation after the flame was removed.
HS-966 (Back Side)	9	2	0.4	It took 5 to 7 seconds before a flame developed on the sample. Very little propogation after the flame was removed.

Material	Vertical Flame Test			Observations
,	Flame Time (seconds)	Glow Time (seconds)	Char Length (inches)	
Aluminized Glass	5	0	2.5	White ash residue. Only silicone rubber coating damaged.
HS-966	30	3 .	1.4	Slow burning, low intensity flame.

It can be seen that the HS-966 samples supported a flame for a longer period of time after removal from the source of flame. The degradation of these samples, however, was limited to a smaller area. B.F. Goodrich Company sponsored development efforts are continuing in the area of improvement of the flame retardant properties of the constituent silicone rubber compounds.

SECTION XIII

CONCLUSIONS AND RECOMMENDATIONS

Ten thermal radiation shields were constructed from HS-966 to fit the hardware of a F-100F pilot's protective hood package, Part No. 243-53090-31 FSN 1680-620-5477. Uncured materials were laid up on a fiberglass building form and vacuum cured. The reflective RTV coating was then sprayed over the shields using conventional paint spraying equipment. After post curing, snap fasteners and reinforcing wires were added to the shields. Small repairs were easily made during final finish operations using body ply materials which had been trimmed from the edges of the shields. These repairs were made with RTV adhesives. The shields were given the B.F. Goodrich Part No. 5A1719.

The shields were delivered to Wright-Patterson Air Force Base for in-service evaluation. Sufficient 100 denier fabric was available for production of four shields from Type I, HS-966. The remaining six were fabricated from HS-966 Type II.

It was determined that fabrication of articles from the newly developed materials is economically feasible. Full-scale factory processing of HS-966 would result in a material cost comparable to aluminized glass.

It would be recommended that further studies be performed to improve the design of the F-100F hood package. The shields could be stowed in a much smaller space if design of the supporting arches and roller and track mechanism were improved.